


**ICF International / Laboratory Data Consultants**

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MEMORANDUM

TO: Chris Lichens, Remedial Project Manager
Site Cleanup Section 4, SFD-7-4

THROUGH: Rose Fong, ESAT Task Order Manager (TOM) RF
Quality Assurance (QA) Program, MTS-3

FROM: Doug Lindelof, Data Review Task Manager 
Region 9 Environmental Services Assistance Team (ESAT)

ESAT Contract No.: EP-W-06-041
Technical Direction Form No.: 00105041 Amendment 7

DATE: January 8, 2008

SUBJECT: Review of Analytical Data, **Tier 2**

Attached are comments resulting from ESAT Region 9 review of the following analytical data:

Site:	Omega Chem OU2
Site Account No.:	09 BC LA02
CERCLIS ID No.:	CAD042245001
Case No.:	Not Provided
SDG No.:	IPI0104
Laboratory:	Test America Analytical Testing Corp.
Analysis:	n-Nitrosodimethylamine (NDMA)
Samples:	6 Water Samples (see Case Summary)
Collection Date:	September 1, 2006
Reviewer:	Santiago Lee, ESAT/Laboratory Data Consultants (LDC)

This report has been reviewed by the EPA TOM for the ESAT contract, whose signature appears above.

If there are any questions, please contact Rose Fong (QA Program/EPA) at (415) 972-3812.

Attachment

SAMPLING ISSUES: ☒ Yes ☐ No

Data Validation Report – Tier 2

Case No.: Not Provided
SDG No.: IPI0104
Site: Omega Chem OU2
Laboratory: Test America Analytical Testing Corp.
Reviewer: Santiago Lee, ESAT/LDC
Date: January 4, 2008

I. CASE SUMMARY

Sample Information

Samples: OC2-MW20C-W-0-234, OC2-MW20B-W-0-235,
OC2-MW20A-W-0-236, OC2-MW20A-W-1-237,
OC2-MW9B-W-0-238, and OC2-MW9A-W-0-239
Concentration and Matrix: Low Concentration Water
Analysis: NDMA (GC/MS/MS CI)
Method: EPA Method 1625 Modified
Collection Date: September 1, 2006
Sample Receipt Date: September 1, 2006
Extraction Date: September 8, 2006
Analysis Date: September 13 and 14, 2006

Field QC

Field Blanks (FB): Not Provided
Trip Blanks (TB): Not Provided
Equipment Blanks (EB): Not Provided
Background Samples (BG): Not Provided
Field Duplicates (D1): OC2-MW20A-W-0-236 and OC2-MW20A-W-1-237

Laboratory QC

Method Blanks & Associated Samples:
6I08056-BLK1: (NDMA) All samples

Tables

1B: Data Qualifier Definitions for Organic Data Review

Sampling Issues

The chain of custody (COC) form did not specify the sample to be used for laboratory quality control (QC). The laboratory did not perform matrix spike/matrix spike/duplicate (MS/MSD) analysis for this SDG. Consequently, the matrix-specific accuracy and precision could not be evaluated.

Additional Comments

As directed by the EPA TOM, a Tier 2 data review was performed (review all QC results and calibrations, minus calculation check). A Table 1A is not requested.

For the 09/13/06 and 09/14/06 calibration verifications, the following information are missing in the data package: daily log summary, QA-QC check report with internal standard (IS) areas, daily midpoint check summary, and raw data.

Decafluorotriphenylphosphine (DFTPP) was not analyzed. Since NDMA is analyzed by the chemical ionization (CI) technique, no adverse effect is expected.

This report was prepared in accordance with the following documents:

- ESAT Region 9 Standard Operating Procedure 901, *Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Volatile and Semivolatile Data Packages*;
- EPA Method 1625C, *Semivolatile Organic Compounds by Isotope dilution GC/MS*, June 1989; and
- USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, October 1999.

II. VALIDATION SUMMARY

The data were evaluated based on the following parameters:

<u>Parameter</u>	<u>Acceptable</u>	<u>Comment</u>
1. Holding Time/Preservation	Yes	
2. GC/MS and GC Performance	N/A	
3. Initial Calibration	Yes	
4. Continuing Calibration	N/A	
5. Laboratory Blanks	Yes	
6. Field Blanks	N/A	
7. Labeled Compound	No	C
8. Matrix Spike/Matrix Spike Duplicates	N/A	
9. Laboratory Control Samples/Duplicates	Yes	
10. Internal Standard	N/A	
11. Compound Identification	N/A	
12. Compound Quantitation	No	A, B
13. System Performance	N/A	
14. Field Duplicate Sample Analysis	Yes	

N/A = Not Applicable

III. VALIDITY AND COMMENTS

A. The following detected result is qualified as estimated and should be flagged "J".

- NDMA in sample OC2-MW20A-W-1-237 (below the practical quantitation limit)

Results below the practical quantitation limits (PQLs) are considered to be qualitatively acceptable, but quantitatively unreliable, due to the uncertainty in analytical precision near the limit of detection.

- B. The laboratory reported the NDMA sample practical quantitation limit (PQL) as 0.0019 ug/L. However, the area for low standard is only 843 for the initial calibration (see attached quantitation report, p. 26 in data package). Furthermore, the area is only 2342 for the concentration of 0.0042 ug/L in sample OC2-MW20A-W-1-237 (see attached quantitation report, p. 119 in data package). In the reviewer's professional judgment, the sample PQL should be raised to 0.01 ug/L; non-detected sample results should be reported as 0.01U.
- C. The laboratory did not spike the samples and method blanks with a labeled compound (i.e., surrogate; see Method 1625C Sections 6.8, 10.2.1.3, and 10.2.3.2 and Figure 4). Consequently, the extraction efficiency (surrogate recovery) cannot be evaluated. The NDMA-d6 spiked by the laboratory was used as an internal standard.

TABLE 1B

DATA QUALIFIER DEFINITIONS FOR ORGANIC DATA REVIEW

The definitions of the following qualifiers are prepared according to the document, "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," October 1999.

- U The analyte was analyzed for but was not detected above the reported sample quantitation limit.
- L Indicates results which fall below the Contract Required Quantitation Limit. Results are estimated and are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in the analytical precision near the limit of detection.
- J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- NJ The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- UJ The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Quantitation Report (Not Reviewed)

Data File : C:\MSDCHEM\1\DATA\06SEP12\NNA001.D
 Acq On : 12 Sep 2006 2:44 pm
 Sample : 1PPB WATER ICAL STD# 6060243
 Misc : n-Nitrosamines Water ICAL
 MS Integration Params: rteint.p
 Quant Time: Sep 12 17:53 2006

Vial: 3
 Operator: DF/AI
 Inst : gcms37
 Multiplr: 1.00

Quant Results File: C6I12NWA.RES

Quant Method : C:\MSDCHEM\1\METHODS\C6I12NWA.M (RTE Integrator)
 Title : Nitrosamine Water ICAL 9/12/06, Preextraction IS
 Last Update : Tue Sep 12 17:53:07 2006
 Response via : Initial Calibration
 DataAcq Meth : C6H02NWA

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
1) NDMA-D6	10.78	81	3840	10.00	PPB	0.00
4) NDPA-D14	15.22	145	1282	10.00	PPB	0.00
						Qvalue
2) NDMA	10.73	92	843	0.61	PPB	98
3) NDEA	12.97	120	386	0.30	PPB	89
5) NDPA	15.18	148	180	0.81	PPB	71
6) NPYR	16.45	118	124	0.54	PPB	97

(#) = qualifier out of range (m) = manual integration
 NNA001.D C6I12NWA.M Tue Sep 12 17:53:21 2006

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Quantitation Report (Not Reviewed)

Data File : C:\MSDCHEM\1\DATA\06SEP13\C0913031.D
 Acq On : 13 Sep 2006 8:22 pm
 Sample : IPI0104-04
 Misc : WATER 1L/1mL ----- Batch 6I08056
 MS Integration Params: rteint.p
 Quant Time: Sep 14 7:30 2006

Vial: 7
 Operator: DF/AI
 Inst : gcms37
 Multiplr: 1.00

Quant Results File: C6I12NWA.RES

Quant Method : C:\MSDCHEM\1\METHODS\C6I12NWA.M (RTE Integrator)
 Title : Nitrosamine Water ICAL 9/12/06, Preextraction IS
 Last Update : Tue Sep 12 17:55:56 2006
 Response via : Initial Calibration
 DataAcq Meth : C6I12NWA

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
1) NDMA-D6	10.77	81	2673	10.00	PPB	0.00
4) NDPA-D14	15.22	145	2716	10.00	PPB	0.00
Target Compounds						
2) NDMA	10.72	92	2342	4.45	PPB	88
3) NDEA	12.97	120	49	0.18	PPB	72
5) NDPA	15.18	148	7946	23.23	PPB	89
6) NPYR	16.45	118	27	0.11	PPB	1

Qvalue

(#) = qualifier out of range (m) = manual integration

C0913031.D C6I12NWA.M Thu Sep 14 07:30:08 2006

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